



82966LMB
Customer No. 01333

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

Christine J. Landry-Coltrain, et al

SMALL POROUS POLYESTER
PARTICLES FOR INKJET USE

Serial No. 10/028,130

Filed 20 December 2001

Commissioner for Patents
P.O. Box 1450
Alexandria, VA. 22313-1450

Group Art Unit: 1774

Examiner: Pamela R. Schwartz

I hereby certify that this correspondence is being deposited today with the United States Postal Service as first class mail in an envelope addressed to Commissioner For Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

Christine Polhurst
Christine Polhurst

November 23, 2004
Date

SECOND DECLARATION UNDER RULE 132

1. I, Christine J. Landry-Coltrain, state that I am a resident of Fairport, N.Y., in the county of Monroe and am a citizen of the United States. I obtained a Bachelor of Science degree in McGill University from Montreal, Canada in 1980. I also have a Ph.D. degree from the University of Wisconsin in Madison Wisconsin in 1985, with a focus on polymer science. I have been an employee of Eastman Kodak Company (hereinafter referred to as Kodak) since 1985. I have been assigned to work in research and development in areas relating to polymer science, such as polymer blends and composites, and media development, such as inkjet and thermal media, and studies relating to the physical properties of polymers.
2. I am one of the co-inventors of U.S. Serial No. US 10/028,130.
3. I have read the Office Action issued on July 29, 2004 and I am familiar with the references cited therein.

4. The following particles evidences the functionality of the present invention utilizing different materials contained in the category of polyesters that is now claimed.
5. Two bead preparations were made: PE-12: Small beads of invention and PE-11: Large beads not of invention
6. Preparation of Large beads (PE-11):

An organic phase consisting of 90.00 g Fineclad® 385 (a nonionic, oil-soluble, unsaturated aliphatic polyester resin obtained from Reichhold Co.), 7.2 ml hexadecane, 1.80 g AIBN, 45.0 g divinylbenzene (mixture of *m* and *p* isomers, 80% with remainder being ethylstyrene) and 45.0 g chloromethylstyrene (mixture of *m* and *p* isomers) dissolved in 60180.0 g toluene was prepared. An aqueous phase consisting of 14.4 g dodecanethiol-endcapped acrylamide oligomer (prepared exactly as described in U.S. Patent No. 6,127,453, col. 9, lines 40-55) dissolved in 1080.0 g deionized water was combined with the organic phase in a 2L beaker and the mixture was homogenized for 10 minutes using a Silverson L4R mixer at the highest speed setting. The resulting dispersion was transferred to a 3-neck round bottom flask outfitted with a condenser, nitrogen inlet, and mechanical stirrer, bubble degassed with nitrogen for 10 minutes, and was heated for 16 hours at 70°C in a constant temperature bath under a positive pressure of nitrogen. 26.3 g N,N-dimethylethanolamine was added and the dispersion was allowed to stir at 80°C for 24 hours. The resulting dispersion of quaternized particles was subjected to rotary evaporation until the collected condensate came over as one phase. The dispersion was filtered through four ply cheesecloth and was purified further by diafiltration with 8 volumes of water through a 100K cutoff membrane using a Millipore Amicon® ultrafiltration apparatus and concentrated to 21.3% solids. The mean particle size of the washed dispersion was determined to be 3.26 µm (CV = 40.04%, Proportion = 100 %) using a Horiba LA-920® particle size analyzer.

7. Small beads (PE-12)

The same aqueous and organic phases described for PE-11 were prepared and combined. The combined phases were emulsified by a two-step process consisting homogenization for 5 minutes using a Silverson L4R mixer followed by passage twice through a microfluidizer. The emulsified reaction mixture was transferred to a 3-neck round bottom flask outfitted with a condenser, nitrogen inlet, and mechanical stirrer, bubble degassed with nitrogen for 10 minutes, and was heated for 16 hours at 70°C in a constant temperature bath under a positive pressure of nitrogen. 26.3 g dimethylethanolamine was added and the reaction was stirred overnight at 70°C. The resulting dispersion of quaternized particles was subjected to rotary evaporation until the collected condensate came over as one phase. The dispersion was filtered through four ply cheesecloth and was purified further by dialysis for 16 hours using 12-14K cutoff dialysis tubing. The resulting dispersion was freeze-dried to obtain 142.5 g of a fine white powder which was easily redispersible in water. The particle size was found to be 0.371 μm (CV = 29.11%, Proportion = 100 %) using a Horiba LA-920® particle size analyzer.

8. Preparation of Element using P-12 small invention beads of Fineclad polyester

A coating composition was prepared from 10.2 wt. % of porous particles PE-12, 1.8 wt. % poly(vinyl alcohol), PVA, (Gohsenol® GH-17 from Nippon Gohsei Co.), and 88 wt. % water. [The relative proportions of porous polyester particle to PVA are therefore 85/15 by weight]. The solution was coated onto a base support comprised of a polyethylene resin coated photographic paper stock, which had been previously subjected to corona discharge treatment, using a calibrated coating knife, and dried to remove substantially all solvent components to form the ink receiving layer. The thickness of the dry ink receiving layer was measured to be about $25 \pm 2 \mu\text{m}$.

9. Preparation of Control Element using P-11 large beads of Fineclad polyester
- A coating composition was prepared from 71.76 wt. % of the PE-11 aqueous dispersion, 2.7 wt. % poly(vinyl alcohol), PVA, (Gohsenol® GH-17 from Nippon Gohsei Co.), and 25.53 wt. % water. [The relative proportions of porous polyester particle to PVA are therefore 85/15 by weight]. The solution was coated onto a base support comprised of a polyethylene resin coated photographic paper stock, which had been previously subjected to corona discharge treatment, using a calibrated coating knife, and dried to remove substantially all solvent components to form the ink receiving layer. The thickness of the dry ink receiving layer was measured to be about $38 \pm 2 \mu\text{m}$.
10. Gloss Assessment of Coated Elements

| Element | Cyan D-max | 60° gloss | Mean diameter (micron) | Proportion (%) |
|-----------------|------------|-----------|---------------------------|----------------|
| P-12 | 1.9 | 47 | 0.37 | 100 |
| Control P-11 | 1.4 | 2.4 | 3.3 | 100 |

The above results show that high surface gloss for the element containing porous polyester particles can only be achieved with the incorporation of porous polyester particles that have a mean diameter of less than 0.5 micrometers, compared to the control elements having only porous polyester particles that have a mean diameter of greater than 0.5 micrometers.

11. I further declare that all statements made herein of my own knowledge are true and that the statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent resulting therefrom.

Date: Nov-19-2004


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DECLARATION UNDER RULE 132

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2. I am one of the co-inventors of U.S. Serial No. US 10/028,130.
3. I have read the Office Action issued on November 19, 2003 and I am familiar with the references cited therein.

4. Element 1 (prepared from particles showing a mean diameter of 0.356 microns, see Table 4 (PE-1)) shows a gloss value of 36. Control Element C-1 (prepared from particles showing a mean diameter of > 0.897 microns, see Table 4 (PE-7)) shows a gloss value of 3, much less than Element 1. All the particles used in Elements 1-6 have their most prevalent size (mode with greater than 50 % (proportion)) less than 0.5 microns. These all show high gloss. Control examples C-1 and C-2 show lower gloss because the particles used are mostly > 0.5 microns.
5. With respect to Claims 29, 30, and 31, the majority of our particles have a mean diameter less than 0.5 microns. Claims 29 and 31 just say that there could be some particles that are larger. This is illustrated in Element 6 (prepared from particles PE-6; see Table 4), for example, where the particles used have predominantly (82.1%) a mean diameter 0.43 microns but also have a component fraction (17.9%) that have a larger size of 5.86 microns.
6. The particles from Maeda are described as having a mean diameter > 0.5 microns, thus indicating they are monomodal (they teach this) and do not have a majority of particles less than 0.5 microns.
7. I further declare that all statements made herein of my own knowledge are true and that the statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent resulting therefrom.

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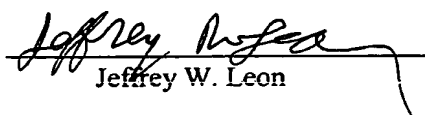
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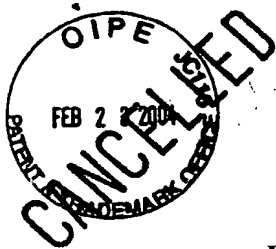
SECOND DECLARATION UNDER RULE 132

1. I, Jeffrey W. Leon, state that I am a resident of Rochester, N.Y., in the county of Monroe and am a citizen of the United States. I obtained a Bachelor of Science degree in Chemistry from State University of New York (SUNY) in Albany, NY in 1989. I was an employee of Schenectady Chemicals in Schenectady, New York in 1989 as a resins chemist. I received a PhD in Chemistry from the University of Rochester, Rochester, NY in 1994, relating to photodegradation of polyester coatings. I did Postdoctoral work at Cornell University, in Ithaca, New York, from 1994-1996, relating to synthesis of dendrimeric polymers. I have been an employee of Eastman Kodak Company (hereinafter referred to as Kodak) since 1996. I have been assigned to work in research development relating to synthesis of latex polymer particles and beads, thermoreactive polymers, inkjet receiver media, polyester synthesis, and water-borne coatings.
2. I am one of the co-inventors of U.S. Serial No. US 10/028,130.
3. I have read the Office Action issued on November 19, 2003 and am familiar with the references cited therein.

4. For the method of preparation of the polyester-containing particles of this invention, different emulsification methods are used. It is the choice of emulsification method, which, of all of the reaction parameters, most strongly influences the resulting particle size. Hence different emulsification methods are commonly used to make particles which differ in particle size.
5. Higher shear methods, such as microfluidization, will typically give smaller particles than, for example, homogenization using a Silverson Mixer.
6. After emulsification, a dispersion results which consists of droplets of the ethylenically unsaturated monomer(s) (in this case, divinylbenzene), the polyester, and the porogenic solvent (in this case, toluene). As the crosslinking reaction is initiated, the droplet becomes a porous microsphere. The composition of the porous microsphere would not be expected to be different if it was formed from a smaller or larger droplet.
7. The microsphere should be the same regardless of the type of emulsification used except that the size will be different. Any scientist trained in the art would expect the composition and porous morphology of the particles to be the same regardless of the type of emulsification used. Only the particle size is affected.
8. To achieve the variety of particle sizes necessary to evaluate the present invention, different emulsification techniques were utilized.
9. I further declare that all statements made herein of my own knowledge are true and that the statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent resulting therefrom.

Date: 11/18/04


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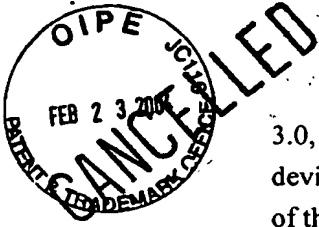
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2. I am one of the co-inventors of U.S. Serial No. US 10/028,130.
3. I have read the Office Action issued on November 19, 2003 and am familiar with the references cited therein.

4. It would have been obvious to one of ordinary skill in the art to treat the surface of the medium of the reference in order to obtain a desired level of gloss." In my opinion, it was not obvious to one of ordinary skill in the art to use particles of less than 0.5 to produce a glossy surface. As explained in the following paragraphs 5-7.
5. It is known in the arts of coating and colloid science that smaller particles are more prone to flocculation during drying processes than larger ones. Flocculation results in smaller particles forming larger clusters, which impart larger feature sizes to a coating. This very often results in less gloss.
6. It is known in the arts of coating and colloid science that coatings of smaller particles will build up large capillary stresses, which will result in mudcracking (and thus loss of gloss) of the coatings. Larger particles are less prone to this phenomenon.
7. Smaller particles often necessitate the use of larger amounts of binder in the coating, which will decrease ink absorption speed. It is well known that the increased surface area of smaller particles typically require more binder to cover the surface of the particles.
8. The cited reference Maeda teaches *away* from particles with a size of < 0.5 microns. See paragraph [00061] "The volume-average particle size D of the particles used in the present invention is in the range of 0.5-100 microns, preferably in the range of 1-50 microns, more preferably in the range of 2-25 microns.... If the volume-average particle size is too small, binding of the particles becomes difficult."
9. The calendaring mentioned by the Examiner involves the application of pressure. Porous particles can be crushed during calendaring so this is often not an option. Calendaring requires an extra step, which is not desirable and may not even be feasible in a production process. Calendaring teaches nothing about particle size selection, which produces a glossy surface.
10. In paragraph [0006], Maeda states that the polyester resin is "...obtained by condensing a polyhydric acid alcohol component and a polybasic carboxylic acid component, preferably 5 nmole% or greater of an unsaturated polybasic carboxylic acid." In paragraph [0009], Maeda gives preferred amounts of

unsaturated polybasic carboxylic acids in the polyester resin. The percentages of carboxylic acid monomer, which compose the polyester resin, are unrelated to the final acid number and the acid number cannot be calculated from these percentages.

11. When polyester resins are prepared, the carboxylic acid groups of the polybasic carboxylic acid monomers are reacted with alcohols to form esters. The acid number is an indication of the amount of acid units, which are left unreacted (ie. which are not converted to esters). Thus without knowing the extent of conversion of the polymerization reaction, the molecular weight of the polyester resin, and the ratio of acid units to alcohol units during the polymerization reaction, and the extent to which acid units were destroyed or formed during side reactions, it is impossible to calculate the acid number of the polyester resin.
12. Incidentally, if the polyester recipes given in the examples of the present invention were carried out to their theoretical maximum extent of reaction, the acid numbers would be ZERO for each.
13. The Examiner states "...claims 29-31 which relate to particle size distributions. Since applicants indicate that their particles may be a component of a system of particles, it would appear that all of these claim limitations may be met by subdividing the particles disclosed by the prior art reference into different categories, so that a peak in particle size distribution is formed as set forth in claim 29 or 31 in one case, or so that there is a standard deviation of particle sizes as set forth by claim 30 in another. The particles of Maeda cannot simply be divided into different categories, so that a peak in particle size distribution is formed as set forth in claim 29 or 31. Claims 29 and 31 refer to the "mode." A mode is a "hump" in a distribution curve. Mathematically, it is a point in a curve where the first derivative equals zero. A gaussian distribution will have only one mode. A multimodal distribution will have multiple points on the curve where the first derivative equals zero. The modality of a distribution is a critical feature of particles, which can influence many different properties. Maeda makes no mention at all of the modality of his distributions.
14. Claim 30 talks about our distribution as *a whole*. To satisfy the conditions of claim 30, the particles of Maeda would have a coefficient of variation of 0.3 to



3.0, that is, 30-300%, as the standard deviation equals the standard deviation/mean. The coefficient of variation is a measure of the "narrowness" of the distribution. At the end of paragraph [0006], Maeda states "Also, the variation coefficient (*same thing as CV*), a value obtained by dividing the standard deviation by the average value, should be 30% or less (*ie. 0.3*), preferably 20% or less, more preferably..." Thus Maeda teaches away from the conditions of Claim 30. Maeda says the distribution should be narrow. The present invention says the distribution should not be so narrow.

15. Furthermore, the distribution specified by Meada (having a $CV < 30\%$) is a "narrow" distribution. The distribution, which fulfills the conditions of claim 30 (having CV greater than or $= 30\%$), is a "broader" distribution than that of Maeda. Thus it is impossible to "take a cut" of Maeda's distribution (either literally or theoretically) which will satisfy the specifications of Claim 30. In other words, you cannot "subdivide" a narrow peak into a portion which is wider than the parent.
16. I further declare that all statements made herein of my own knowledge are true and that the statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent resulting therefrom.

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